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(54) **Water-borne treatment compositions for porous substrates.**

(57) Disclosed herein is a treating composition for a porous substrate in the form of an oil-in-water dispersion comprising a dispersed phase of an organic substrate penetrating solvent, a water repellent, a substrate preservative, a hydrocarbon resin, and a stabilizing surfactant, and a method of preparing the same by particularization with a dispersing means.

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Background of the Invention

The invention is directed to an aqueous treatment composition and a method for rendering porous substrates water repellent and for preserving porous substrates utilizing the aqueous treatment composition.

Generally, the art-known porous substrate treatment compositions such as wood treatment compositions are organic solvent-borne compositions. For example, U.S. Patent Nos. 4,360,385 and 4,404,239 are directed to solvent-borne wood treatment compositions containing olefin and paraffin materials containing a substantial proportion of compounds containing from about 20 to 24 carbon atoms and, optionally, additives such as wood preservatives, alkyd resins and organic ionizable compounds. It is disclosed in the aforesaid United States patents that such art-known organic solvent-borne wood treatment compositions can provide wood with water-repellency at an efficiency level of at least about 40 percent, preferably at least about 50 percent and more preferably at least about 60 percent as determined by the N.W.M.A. Swellometer Test described in detail in a publication identified as NWMA-M-2-81, published by the National Woodwork Manufacturers Association.

Increasing environmental concerns have generated an ever increasing need to develop treatment compositions with lower levels of organic solvents. One approach to reducing the organic solvent content investigated by the present inventors involves the utilization of an aqueous treatment composition. However, heretofore water-borne wood treatment compositions have not been able to provide nearly the level of water-repellency to wood as organic solvent-borne treatment compositions. For example, N.W.M.A. Swellometer Test NWMA-M-2-81, Sec. 3.2.1 concerning the method of treating wood for the purposes of the test discloses that the five test specimens (immersed) in the formulation to be tested have an immersion time for solvent-based formulations of 30 seconds and immersion time for water-borne formulations of 3 minutes. In part, the difficulty in water-repellency is believed to be due to the inefficient penetration of the wood by the water-borne formulation. Penetration of wood by water is inefficient because water, upon penetrating wood, swells the wood and hinders further penetration of the wood. Hence, other ingredients are hindered from penetrating the wood in a short exposure time. By the present invention, there is provided an aqueous wood-treating composition having penetration and water-repellency that is comparable to that of solvent-borne compositions.

Summary of the Invention

In accordance with the foregoing, the present invention encompasses an aqueous treatment composition for porous substrates in the form of a dispersion comprising:

(A) from about 5 to 80 percent by weight of a dispersed phase containing 10 to 90 percent by weight of a porous substrate-penetrating organic solvent, from about 2.0 to 90 percent by weight of a water-repellent selected from the group consisting of an alpha-olefin or paraffin blend having 20 to 24 carbon atoms, from about 0 to 20 percent of a porous substrate preservative, and from 0.10 to 10 percent by weight of a water-soluble surfactant, and

(B) from about 30 to 90 percent by weight water; wherein the dispersed organic phase is present in said aqueous treatment composition in an amount insufficient to cause the dispersed organic phase to become a continuous organic phase.

The aqueous treatment composition can be initially prepared in the form of a concentrate, which can be thinned with water with or without supplemental ingredients being added thereto. As an aqueous wood treatment, the composition of this invention is capable of providing a wood substrate with water repellency at a level of at least 40 percent and up to 60 percent or higher as determined by NWMA-M-2-81 modified in that the immersion time for said aqueous wood-treatment composition for NWMA-M-2-81 shall be 30 seconds instead of 3 minutes.

Also encompassed by the invention is a method for making the dispersions and coating of this invention preferably by microfluidization. Further encompassed by the invention is a method of treating porous substrates such as wood which comprises applying the water-repellant composition of this invention to the wooden substrate followed by drying the wooden substrate. The resultant treated wooden substrate can be optionally coated or cladged. While the treatment composition is described herein with particularity to wood treating compositions, the present invention encompasses treating compositions for other porous substrates such as textiles and cements.

Detailed Description of the Invention

As set forth hereinabove, the aqueous treating composition of the invention is an oil-in-water dispersion.

By the term "oil-in-water dispersion" is meant that the dispersed phase herein is an oil and the continuous phase is water or an aqueous medium.

The dispersed phase of the oil-in-water dispersion of this invention is in the range of about 5 to 80 and preferably 10 to 70 percent by weight based on total weight of the dispersion. The dispersed phase
 5 comprises a solvent medium containing a mixture of solvents which are substrate-penetrating solvents, and further comprises a water repellent. The dispersed phase contains from about 5 to 90 percent by weight of a porous substrate penetrating organic solvent, and from about 1.5 to 90 percent of the water repellent.

The useful wood penetrating solvents for example, are selected on the basis that they are generally non-polar and do not appreciably associate with the wood components, say, by hydrogen bonding.
 10 Illustrative but non-limiting examples of the solvents can be xylene, toluene, mineral spirits, naphtha, ketones and a mixture thereof.

The water repellent of the dispersed phase comprises an alpha olefin or paraffin blend having about 20 to 24 carbon atoms or a mixture thereof with the alpha olefin blend being preferred. The individual cuts of alpha olefin having about 20 to 24 carbon atoms are useful, though such cuts are not readily available and
 15 are generally less preferred for this reason. The paraffin material can only be used as a blend of compounds as described below, it being found that individual paraffins do not provide the desired water-repellency results. The olefin and paraffin blends are characterized by consisting essentially of a narrow range of carbon compounds. That is, while paraffins are well known and have been used extensively heretofore, such materials are blends of a wide range of different carbon chain length materials. These
 20 commonly used paraffin blends are difficult to coat over and do not provide the good water-repellency efficiencies of the olefin and paraffin compounds described herein. It has been found that narrow molecular weight blends (cuts) of olefin and/or paraffin provide good water-repellency to wood. At least about 50 percent of the olefin blend or paraffin blend consists essentially of compounds having from 20 to 24 carbon atoms. Preferably, the olefin blend and paraffin blend contain at least about 70 percent and, more
 25 preferably, at least about 90 percent of the compounds having between 20 and 24 carbon atoms. The most preferred blend contains at least about 96 percent of the compounds having 20 to 24 carbon atoms and less than about 3 percent of the compounds having less than 20 carbon atoms and less than about 1 percent having more than 24 carbon atoms.

Substituted and unsubstituted hydrocarbon resins particularly alkyd resins have conventionally been
 30 used in wood-treating compositions to provide a degree of water-repellency and to aid holdout of subsequently applied materials. The alkyd resins are polyesters of polyhydroxyl alcohols and polycarboxylic acids chemically combined with various drying, semi-drying and nondrying oils in different proportions. Thus, for example, the alkyd resins are made from polycarboxylic acids such as phthalic acid, maleic acid, fumaric acid, isophthalic acid, succinic acid, adipic acid, azelaic acid, sebacic acid as well as from
 35 anhydrides of such acids where they exit. The polyhydric alcohols which are reacted with the polycarboxylic acid include glycerol, trimethylolethane, trimethylolpropane, pentaerythritol, sorbitol, mannitol, ethylene glycol, diethylene glycol and 2,3-butylene glycol. The alkyd resins are produced by reacting the polycarboxylic acid and the polyhydric alcohol together with a drying, semi-drying or non-drying oil in proportions depending upon the properties desired. The oils are coupled into the resin molecule by esterification during
 40 the manufacturing and become an integral part of the polymer. The oil is fully saturated or predominately unsaturated. The fully saturated oils tend to give a plasticizing effect to the alkyd, whereas the predominately unsaturated oils tend to crosslink and dry rapidly with oxidation to give more tough and durable alkyd resins. Suitable oils include coconut oil, fish oil, linseed oil, tung oil, castor oil, cottonseed oil, safflower oil, soybean oil and tall oil. Various proportions of the polycarboxylic acid, polyhydric alcohol and oil are used
 45 to obtain alkyd resins of various properties. Phenolic modified hydrocarbon resins such as NEVILLAC 10[®] XL, available from Neville Chemical Co. have also been found to be particularly useful in providing water repellency and hold out.

Many known compounds which are useful as substrate preservatives for some porous substrate such as wood. Examples of wood preservatives include carbamates such as 3-iodo-2-propyl butyl carbamate;
 50 organic tin compounds, e.g., triphenyl and tributyl tin oxide; chlorinated compounds, e.g., tri-, tetra- and pentachlorophenol, mono- and dichloro naphthalenes, and metal naphthenates, e.g., zinc and copper naphthenates, and quaternary ammonium compounds. The preservative content can be from about 0 to 20 percent and typically 0.5 to 20 percent by weight of the dispersed phase.

Other conventional additives can be included in the treating composition of this invention. For example,
 55 surface modifiers, insecticides, biostatic agents, biocides or a mixture thereof can be employed. Pigments can also be employed herein.

In the process of this invention, the intended ingredients of the dispersion, including the optional ingredients, are dispersed in an aqueous medium with a dispersing means such as a homogenizer.

Typically, a mixture of the ingredients is adapted to a form that will admit it to the dispersing means. Illustratively, the mixture is in the form of a solution or is dissolved with a solvent or a mixture of solvents and then introduced into an aqueous medium in the presence of a stabilizer.

Typically, the solvent employed here is the same as the aforescribed substrate-penetrating solvent. Preferably, a cosolvent is employed as well. It is believed that the (primary) solvent which is preferably a non-polar solvent dissolves the mixture and the cosolvent which is preferably polar enhances the stability of the resultant composition. The dissolved mixture is mixed into an aqueous medium in the presence of the stabilizer. Illustratively, the dissolved mixture is introduced into and thoroughly mixed with an aqueous medium containing a stabilizer which is typically a surfactant or a combination of surfactants. The resultant mixture can be described as an emulsion.

The stabilizer can be a surfactant which is typically an anionic and/or a nonionic surfactant, preferably in combination with an alcohol containing about 10 to 16 carbon atoms (C₁₀-C₁₆ alcohol). Specific but non-limiting examples of the surfactant combinations that can be found in the working examples of the invention. Amounts of about 1 to 10 percent and preferably about 1 to 4 percent by weight resin solids of the anionic surfactant based on the composition can be employed. Amounts of about 1 to 10 percent by weight and preferably about 1 to 6 percent by weight resin solids of the nonionic surfactant or the C₁₀-C₁₆ alcohol based on the composition can be employed. In the preferred embodiment of the invention, the surfactant combination comprises an anionic surfactant and a nonionic co-surfactant in combination with the C₁₀-C₁₆ alcohol. It has been found that such a surfactant combination can improve stability of the dispersions of the invention.

The emulsion can be particulated by subjecting it to stress by a homogenizing means. The mixture is subjected to stress sufficient to produce microparticles which have a mean particle size diameter of about 500 to 3,000 angstroms (Å) and typically about 500 to 1500Å, preferably in monomodal distribution.

A preferred mode of subjecting the mixture to the appropriate stress is by the use of a MICROFLUIDIZER® emulsifier, which is available from Microfluidics Corporation in Newton, Massachusetts. The MICROFLUIDIZER® high-pressure impingement emulsifier is patented in U.S. Patent No. 4,533,254, which is incorporated herein by reference. The device consists of a high pressure (up to 20,000 pounds per square inch "psi") pump and an interaction chamber wherein the emulsification takes place. The pump forces the mixture of ingredients in an aqueous medium into the chamber where it is split into at least two streams which pass at very high velocity through at least two splits and collide resulting in the particulation of the mixture into small particles. Generally, the mixture is passed through the emulsifier once at a pressure between 5,000 and 15,000 psi. Multiple passes can result in smaller average particle size and a narrower range for the particle size distribution. When using the aforesaid MICROFLUIDIZER® emulsifier, stress is applied by liquid impingement as has been described. However, it should be understood that, if desired, other modes of applying stress to the pre-emulsification mixture can be utilized so long as sufficient stress is applied to achieve the requisite particle size distribution, that is, such that, less than 20 percent of the resultant microparticles have a mean diameter greater than 5 microns in the resultant product. Alternate means of applying stress, e.g., the use of ultrasonic energy can be employed.

Stress is described as a force per unit area. Although the precise mechanism by which the MICROFLUIDIZER® emulsifier stresses the pre-emulsification mixture to particulate, it is not thoroughly understood; it is theorized that stress is exerted in more than one manner. It is believed that one manner in which stress is exerted is by shear. "Shear" means that the force is such that one layer or plane moves parallel to an adjacent parallel plane. Stress can also be exerted from all sides as a bulk compression stress. In this instance, stress could be exerted without any shear. A further manner of producing intense stress is by cavitation. Cavitation occurs when the pressure within a liquid is reduced enough to cause vaporization. The formation and collapse of the vapor bubbles occurs violently over a short time period and produces intense stress. Although not intending to be bound by theory, it is believed that both shear and cavitation contribute to producing the stress which particulates the mixture.

The resultant dispersion can be in the form of a concentrate containing from about 5 to 80 percent and preferably from about 20 to 50 percent solids of dispersed phase based on the total weight at ambient temperature. The concentrate is generally further diluted with water to form a ready-to-use dispersion.

Compositions of this invention comprising the dispersion are especially useful when used for treating wood. The compositions can be applied by any conventional method including dipping, flow coating, roll coating, and in a vacuum/pressure apparatus. Generally, the amount of treating composition applied is sufficient to provide the wood with the desired water-repellency. Water repellency of the treated composition can be at a level of at least 40 percent and preferably 50 percent and more preferably 60 percent as determined by NWMA-M-2-81 Swellometer test with an immersion time of 30 seconds. The wood, after being treated, is optionally further processed and then dried.

Further illustrating the invention are the following non-limiting examples.

EXAMPLE 1

5 The following treatment composition of this invention was formulated as follows. In a 1 liter beaker was placed 8.1 percent C₂₀-C₂₄ alpha olefin¹, 13.4 percent hydrocarbon resin², 2.0 percent 3-iodo-2-propynyl butyl carbamate³, 4.1 percent anionic⁴ and 6.1 percent nonionic⁵ surfactants, 4.1 percent alcohol⁶, .284 percent microbiocide⁷, and finally 20.4 percent "Solvesso 150"⁸. The mixture was warmed until all the components formed a homogeneous liquid. The liquid was then introduced into a dispersing apparatus⁹ and
10 water was added until there was a drastic increase in viscosity (the solution had a viscosity in excess of 200 centipoises (cps) at this point, which was its inversion point). The resultant emulsion was a concentrate in the form of an oil-in-water emulsion that could be further diluted with water.

- (1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".
- (2) Available from Neville Chemical as "Nevillac 10° XL".
- 15 (3) Available from Troy Chemical as "Polyphase P-100".
- (4) Available from GAF Corporation as "Alipal CO-436".
- (5) Available from GAF Corporation as "Igepal CO-897".
- (6) Available from Exxon as "Exxal 13".
- (7) Available from ICI Americas as "Proxel CRL".
- 20 (8) Available from Exxon as "Solvesso 150".
- (9) Available from Microfluidics Corporation as "Microfluidizer® emulsifier".

EXAMPLE 2

25 The emulsion from Example 1 was diluted by three parts by volume water to one part by volume concentrate to yield a ready-to-use wood- treating composition. The water repellency of the composition was tested following the NWMA #M-2-81 test method. The wood preservative properties were tested by NWMA Soil Block Test #M-1-81 at Mississippi State University.

The ready to use wood treating composition was as follows:

30

Ingredients	Percent Compositions
Alpha Olefin ¹	2.0
Hydrocarbon Resin ²	3.3
35 Preservative ³	.50
Solvent ⁴	5.0
Surfactant 1 ⁵	1.0
Surfactant 2 ⁶	1.0
Alcohol ⁷	1.5
40 Microbiocide ⁸	.07
Water	85.63

- (1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".
- (2) Available from Neville Chemical as "Nevillac 10° XL".
- 45 (3) Available from Troy Chemical as "Polyphase P-100".
- (4) Available from Exxon as "Solvesso 150".
- (5) Available from GAF Corporation as "Alipal CO-436".
- (6) Available from GAF Corporation as "Igepal CO-897".
- (7) Available from Exxon as "Exxal 13".
- 50 (8) Available from ICI Americas as "Proxel CRL".

Three to five matched wood wafers were dipped in the above composition for a period of 30 seconds as prescribed by the NWMA M-2-81 Swell-O-Meter test procedure. A water-repellency efficiency of 70.1 after five days was obtained.

55 The above composition was sent to Mississippi State University for soil block testing. Summary of the individual percent weight losses of treated southern yellow pine blocks (3.05 x 19 x 19mm) after exposure to brown-rot fungus (*G. trabeum*) for five weeks are stated in the table below.

TABLE 1

5	FORMULA	% CONCEN	BLOCK #	RETENTION (pcf) ¹	W1 ²	W2 ³	% WT. LOSS	VISUAL
		TRATION		BIOCIDE	(g)	(g)		INSPECT.
10	Ex. 1	100.00%	W-21	54.430	1.04	1.04	0.000	ND ⁴
			W-22	51.028	1.10	1.10	0.000	ND
			W-23	53.863	1.10	1.10	0.000	ND
			W-24	67.470	0.93	0.91	2.150	ND
			W-25	69.171	0.94	0.93	1.060	ND
20	Control	-----	W-31	0.000	0.97	0.51	47.42	D ⁵
			W-32	0.000	0.93	0.54	41.94	D
			W-33	0.000	0.95	0.46	51.58	D
			W-34	0.000	0.80	0.39	51.25	D
			W-35	0.000	0.77	0.45	41.56	D

25 (1) pcf stands for pounds per cubic foot.

(2) W1 stands for initial weight in grams (g) of the wood before exposure to the fungus.

30 (3) W2 stands for weight in grams (g) of the wood after exposure to the fungus.

(4) ND stands for "No Decay" upon visual inspection.

35 (5) D stands for "Decay" upon visual inspection.

EXAMPLE 3

40 The following shows the effects of various resins on the water repellency efficiency of the treatment composition. The treatment composition was essentially the same as in Example 1 except that different hydrocarbon resins were employed and the concentrate was diluted to yield the following compositions.

45

50

55

Composition (%)	A	B	C
Alpha Olefin ¹	2.0	2.6	5.0
Hydrocarbon Resin ²	3.3		
Hydrocarbon Resin ³		14.1	
Hydrocarbon Resin ⁴			11.6
Preservative ⁵	.50	.50	.50
Solvent ⁶	5.0	5.0	5.0
Surfactant 1 ⁷	1.0	.50	
Surfactant 2 ⁸	1.0		.50
Alcohol ⁹	1.5	.20	1.5
Microbiocide ¹⁰	.07	.07	.07
Water	85.63	77.03	81.33
Water-repellency	70.1	55.3	64.3

(1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".

(2) Available from Neville Chemical as "Nevillac 10 ° XL".

(3) A dehydrated castor oil short oil alkyd.

(4) A soya oil long oil alkyd.

(5) Available from Troy Chemical as "Polyphase P-100".

(6) Available from Exxon as "Solvesso 150".

(7) Available from GAF Corporation as "Alipal CO-436".

(8) Available from GAF Corporation as "Igepal CO-897".

(9) Available from Exxon as "Exxal 13".

(10) Available from ICI Americas as "Proxel CRL".

EXAMPLE 4

This example shows the importance of the alpha olefin as compared to paraffin wax for water-repellency efficiency. The following formulations were made and tested for water repellency as described in Example 2:

<u>Composition</u>	<u>Water-repellency</u>
Example 1	70.1
Example 1 (with paraffin ¹ instead of alpha olefin)	14.8

(1) From W&F Manufacturing Corporation, Buffalo, New York.

EXAMPLE 5

This example illustrates the efficiency of the treatment with respect to dip time. The composition of Example 1 was formulated and the same procedure was followed for reduction. The water-repellency efficiency measurement was conducted as per NWMA M-81 test except that the employed wafers were dipped for 20 and 30 seconds. The results after 5 days were:

	<u>Composition</u>	<u>Dip Time</u>	<u>Water-repellency</u>
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	Example 1	20 sec.	60.1
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5	Example 1	30 sec.	70.1
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	"Woodlife		
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10	Milltreat F ¹ "	30 sec.	75.8
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(1) A solvent based water repellent wood preservative product available from Beecham Home Improvement Products, Inc.

The results clearly show that even using a short dip time of the water-base material, the water repellency efficiency is good. Also, the data shows that this formulation is comparable in efficiency to a commercially available solvent based, wood water repellent, preservative solution, Woodlife Milltreat F.

EXAMPLE 6

This experimental design used to determine the optimal surfactant levels illustrates the interaction between the surfactant levels and penetration of the preservative. The compositions were formulated as in Example 1 indicated and diluted as in Example 2 to yield the following compositions.

Percent Composition								
Ingredients	A	B	C	D	E	F	G	H
Alpha Olefin ¹	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Hydrocarbon Res. ²	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3
Preservative ³	.50	.50	.50	.50	.50	.50	.50	.50
Solvent ⁴	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Alcohol ⁵	1.0	1.0	1.0	1.0	2.0	2.0	2.0	2.0
Surfactant 1 ⁶	1.0	.50	1.0	.50	1.0	.50	1.0	.50
Surfactant 2 ⁷	.80	.80	1.5	1.5	1.5	1.5	.80	.80
Microbiocide ⁸	.07	.07	.07	.07	.07	.07	.70	.70
Water	86.33	86.83	85.63	86.13	84.63	85.13	85.33	85.83

(1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".

(2) Available from Neville Chemical as "Nevillac 10" XL".

(3) Available from Troy Chemical as "Polyphase P-100".

(4) Available from Exxon as "Solvesso 150".

(5) Available from Exxon as "Exxal 13".

(6) Available from GAF Corporation as "Alipal CO-436".

(7) Available from GAF Corporation as "Igepal CO-897".

(8) Available from ICI Americas as "Proxel CRL".

After a 30 second dip in the treatment, the 1.125 x 1.125 x 12.0 inch pine penetration block were allowed to dry for seven days at room temperature. The blocks were then planed on all four faces until they measure 1.0 x 1.0 x 12.0 inch and then cut into .25 inch thick wafers and crushed for detection of the active ingredient by "Soxhlet" extraction.

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Solution	Depth into wood (inches)	Penetration of IPBC
A	.25	.765ug
	.50	<.02ug
B	.25	.681ug
	.50	<.02ug
C	.25	.682ug
	.50	.841ug
	.75	.634ug
	1.00	1250ug
	1.25	983ug
	1.50	880ug
	1.75	1030ug
D	2.00	460ug
	.25	.538ug
E	.50	.023ug
	.25	.590ug
F	.50	<.020ug
	.25	.612ug
G	.50	<.020ug
	.25	.659ug
H	.50	<.020ug
	.25	.672ug
	.50	<.020ug

The results show that the levels of the various surfactants chosen in sample "C" optimize the penetration of the preservative to a depth far beyond the others.

EXAMPLE 7

This example shows the versatility of the formulation which allows one to change the active ingredient to fit the product requirements. The following emulsions were formulated as in Example 1 and diluted as in Example 2 to yield the following:

Percent Composition			
Ingredient	A	B	C
Alpha Olefin ¹	2.0	2.0	2.0
Hydrocarbon Resin ²	3.3	3.3	3.3
Preservative ³	.50		
Preservative ⁴		1.0	
Preservative ⁵			3.0
Solvent ⁶	5.0	5.0	5.0
Surfactant 1 ⁷	1.0	1.0	1.0
Surfactant 2 ⁸	1.0	1.0	1.0
Alcohol ⁹	1.5	1.5	1.5
Microbiocide ¹⁰	.07	.07	.07
Water	85.63	85.13	83.13

(1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".

(2) Available from Neville Chemical as "Nevillac 10° XL".

(3) Available from Troy Chemical as "Polyphase P-100".

(4) Available from Beckman Laboratories as "Busan 1009".

(5) Available from Sherex Chemicals as "Adogen 432".

(6) Available from Exxon as "Solvesso 150".

(7) Available from GAF Corporation as "Alipal CO-436".

(8) Available from GAF Corporation as "Igepal CO-897".

(9) Available from Exxon as "Exxal 13".

(10) Available from ICI Americas as "Proxel CRL".

The products were stable for at least three months in ambient temperatures and in a 120° hot room.

EXAMPLE 8

This illustration shows the versatility of the formulation which allows one to change the solvent system to fit the product application. The following formulations were formulated as stable samples as in Example 1 and diluted as in Example 2 to yield the following formulations:

Percent Composition						
Ingredient	A	B	D	E	F	G
Alpha Olefin ¹	2.0	2.0	2.0	2.0	2.0	2.0
Hydrocarbon Resin ²	3.3	3.3	3.3	3.3	3.3	3.3
Preservative ³	.50	.50	.50	.50	.50	.50
Solvent ⁴	5.0					
Solvent ⁵		5.0				
Solvent ⁶			5.0			
Solvent ⁷				5.0		
Solvent ⁸					5.0	
Solvent ⁹						5.0
Alcohol ¹⁰	1.0	1.0	1.0	1.0	1.0	1.0
Surfactant ¹¹	1.0	1.0	1.0	1.0	1.0	1.0
Surfactant ¹²	1.5	1.5	1.5	1.5	1.5	1.5
Microbiocide ¹³	.07	.07	.07	.07	.07	.70
Water	85.63	85.63	85.63	85.63	85.63	85.63

(1) Available from Chevron Chemical as a "C₂₀-C₂₄ alpha olefin".

(2) Available from Neville Chemical as "Nevillac 10" XL".

(3) Available from Troy Chemical as "Polyphase P-100".

(4) Available from Exxon as "Solvesso 150".

(5) Xylene available from Ashland Chemical.

(6) Methyl isobutyl ketone available from Exxon.

(7) Cyclohexanone available from Ashland Chemical.

(8) Odorless kerosene available from Aldrich.

(9) Mineral spirits available from Ashland Chemical.

(10) Available from Exxon as "Exxal 13".

(11) Available from GAF Corporation as "Alipal CO-436".

(12) Available from GAF Corporation as "Igepal CO-897".

(13) Available from ICI Americas as "Proxel CRL".

As aforesated, the invention in its broad embodiment encompasses a water-repellant composition for porous substrates other than wood. While the illustrative embodiments of the invention have been described hereinabove with particularity, it will be understood that various modifications of the invention will be apparent to those skilled in the art without departing from the scope or spirit of the invention. Accordingly, the claims directed to the invention are intended to be construed as encompassing all aspects of the invention which would be treated as equivalents by one skilled in the art to which the invention pertains.

Claims

1. An aqueous treating composition for a porous substrate in the form of an oil-in-water dispersion comprising:

(A) from about 5 to 80 percent by weight of a dispersed phase containing about 5 percent to 90 percent by weight of a porous substrate-penetrating organic solvent, from about 1.5 percent to 90 percent by weight of a water-repellent selected from the group consisting of an alpha-olefin or paraffin blend having about 20 to 24 carbon atoms, from about 0 to 20 percent of a porous substrate preservative, and from about 0.10 to 10 percent by weight of a water-soluble surfactant, and

(B) from about 20 percent to 95 percent by weight water; wherein the dispersed organic phase present in said aqueous treating composition in an amount insufficient to cause the dispersed organic phase to become a continuous organic phase.

2. The aqueous treating composition of claim 1 capable of providing a wood substrate with water repellency at a level of at least 40 percent as determined by NWMA-M-2-81 except that the immersion time for said aqueous treating composition for NWMA-M-2-81 is 30 seconds.

3. The aqueous treating composition of claim 1 wherein said dispersed phase is further characterized as having dispersed particles having a mean average diameter of up to 3000Å.

4. The aqueous treating composition of claim 1 comprising from 10 percent to 70 percent by weight of said dispersed phase and from 30 percent to 90 percent by weight water, and further characterized as capable of providing a wood substrate with water repellency at a level of at least 50 percent as determined by modified NWMA-M-2-81 wherein the immersion time for said aqueous wood-treating composition is 30 seconds.
5. A method of treating a porous substrate to render it water repellent comprising:
 - (I) applying to the substrate an aqueous treating composition in the form of an oil-in-water dispersion comprising:
 - (A) from about 5 to 80 percent by weight of a dispersed phase containing 10 to 90 percent by weight of a porous substrate-penetrating organic solvent, from about 2.0 to 90 percent by weight of a water-repellent selected from the group consisting of an alpha-olefin having 20 to 24 carbon atoms, a narrow-cut alpha olefin blend and a narrow-cut paraffin blend, from about 0 to 20 percent of a porous substrate preservative, and from about 0.10 to 10 percent by weight of a water-soluble surfactant, and
 - (B) from about 20 to 95 percent by weight water; wherein the dispersed organic phase is present in said aqueous treating composition in an amount insufficient to cause the dispersed organic phase to become a continuous organic phase; and (II) drying said substrate.
6. The method of claim 5 wherein said aqueous treating composition is capable of providing said substrate with water repellency at a level of at least 40 percent as determined by NWMA-M-2-81 except that the immersion time for said aqueous treating composition for NWMA-M-2-81 is 30 seconds.
7. The method of claim 6 wherein said dispersed phase of said aqueous treating composition is further characterized as having dispersed particles having a mean particle size diameter of up to 3000Å.
8. The method of claim 5 wherein said aqueous treating composition comprises from 10 percent to 70 percent by weight of said dispersed phase and from 90 percent to 30 percent by weight water, and further characterized as capable of providing a wood substrate with water repellency at a level of at least 50 percent as determined by NWMA-M-2-81 except that the immersion time for said aqueous treating composition for NWMA-M-2-81 is 30 seconds.
9. A method of preparing an aqueous treating for a porous substrate in the form of an oil-in-water dispersion comprising particulating in an aqueous medium in the presence of a stabilizer from about 5 to 80 percent by weight of a dispersed phase containing a mixture of 10 percent to 90 percent by weight of a porous substrate penetrating organic solvent, from 2.0 percent to 90 percent by weight of a water-repellent selected from the group consisting of an alpha-olefin or paraffin blend having about 20 to 24 carbon atoms, from about 0 to 20 percent of a porous substrate preservative, and from about 0.10 to 10 percent by weight of a water-soluble surfactant; wherein the dispersed organic phase is present in said aqueous treating composition in an amount insufficient to cause the dispersed organic phase to become a continuous organic phase.
10. The method of claim 9 wherein the mixture is particulated by subjecting it to stress sufficient to produce disposed particles having a mean particle size diameter of 500 to 1500Å.
11. The method of claim 10 wherein stress is applied by liquid to liquid impingement.
12. The method of claim 9 wherein the mixture is particulated with a means comprising a homogenizer.
13. The method of claim 9 wherein the mixture is particulated by passing it through a microfluidizer.
14. The method of claim 13 wherein the mixture is adapted to a form that admits it into the microfluidizer.
15. The method of claim 18 wherein the mixture is adapted to a form for particularization by mixing it with a solubilizing means, followed by emulsifying the resultant solubilized mixture in water in the presence of a stabilizer.
16. The method of claim 15 wherein the solubilizing means comprises a mixture of a polar and a non-polar

solvent.

17. The method of claim 16 wherein the stabilizer is a surfactant comprising a mixture of an anionic and a nonionic surfactant.

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EUROPEAN SEARCH REPORT

Application Number

EP 91 11 3296

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	US-A-3 617 314 (R.E.HILL) * column 5 - column 6; tables I,II,III *** claims 1-14 ** - - -	1-17	B 27 K 3/50
X	US-A-3 281 318 (R.E.STUTZ) * column 7; example 9 *** claims 1-14 ** - - -	1-17	
A	EP-A-0 040 106 (ROBERTS CONSOLIDATED INDUSTRIES) * page 6; table 2 *** claims 1-10 ** - - -	1-17	
A	GB-A-2 128 091 (LAPORTE INDUSTRIES) - - -		
A	CA-A-1 207 952 (CLOVERDALE PAINT & CHEMICALS) - - -		
D,A	US-A-4 360 385 (J.F.GRUNEWALDER) - - - - -		
The present search report has been drawn up for all claims			
Place of search		Date of completion of search	Examiner
The Hague		13 December 91	DALKAFOUKI A.
<div>CATEGORY OF CITED DOCUMENTS</div> <div><div>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention</div><div>E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</div></div>			